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ENGINEERING SERVICE CENTER
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METHOD OF TEST FOR THERMO-APPLIED PAVEMENT STRIPING GRANULES

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Part 10 of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

SCOPE

The procedures used for testing thermo-applied pavement striping granules are described in this method. The material is a mixture of pigmented resin granules and reflective spheres which, when flame sprayed to clean road surfaces, produces a very quick drying, adherent reflectorized stripe.

This test method is divided into the following parts:

1. Grading analysis
2. Glass bead content and grading analysis
3. Ring and ball softening point
4. Reflectance and Yellowness Index
5. Titanium dioxide
6. Bond strength
7. Specific gravity
8. Bleed-through on asphalt
9. Color

10. Safety and Health

PART 1. GRADING ANALYSIS

A. APPARATUS

1. A balance with 0.01-g divisions
2. Standard sieves: 600, 212, and 63- μ m sieves with a bottom pan
3. A sample splitter, sample thief, or other suitable means of obtaining representative samples

B. PROCEDURE

1. Record the weight of each sieve and the bottom pan to the nearest 0.01 g. Assemble the sieves in proper order.
2. From a thoroughly mixed full carton of finished powder, remove two representative samples. Each sample shall weigh 50 g or more.
3. Record the weight of each sample to the nearest 0.01 g and transfer the sample to the nested sieves.
4. After shaking for a suitable time, manually or by machine, separate the sieves and

weigh the material on each sieve to the nearest 0.01 g.

5. Calculate “% retained” per sieve as follows:

$$\% \text{ retained} = [(\text{mass of sieve and retained material} - \text{mass of sieve}) / \text{total sample mass}] \times 100$$

PART 2. GLASS BEAD CONTENT AND GRADING ANALYSIS

A. APPARATUS

1. A balance with 0.01-g divisions
2. Standard sieves: 425, 212, 63 and 53- μm sieves and a bottom pan
3. A sample splitter, sample thief, or other suitable means of obtaining representative samples
4. Solvent (alcohol, aromatic solvent or ketone)
5. An oven capable of maintaining $105 \pm 2^\circ\text{C}$
6. Several round, 500-mL cans with lids

B. PROCEDURE

1. Weigh 100 ± 0.1 g into a 500-mL can.
2. Fill the can with solvent, place a lid on the can and extract the resins by shaking moderately or rolling the sample for 1 h on a paint mill roller until solution is complete. Allow the beads and insoluble fillers to settle and decant the supernatant solution, being careful that the beads are not also decanted. Repeat the extraction until supernatant liquid is clear. Make final extraction with isopropanol only.
3. Add distilled water to the can along with a small amount of detergent. Replace the lid and shake or roll the can for about 30 min. Allow the beads to settle, remove the lid from the can and cut the rim from inside the can.

4. Pour the solution and beads onto the 53- μm sieve and rinse the can thoroughly with distilled water.

5. Wash the beads on the screen with distilled water until they are clear.

6. If a small diameter sieve is used that fits on the balance, dry it in an oven at 105°C . Cool and weigh the sieve. Alternately wash the beads from the sieve into a clean, tared beaker, dry and weigh these materials.

7. $\% \text{ Beads} = [(\text{weight of the beads and the container} - \text{weight of the container}) / \text{weight of the sample}] \times 100$

8. Grade the beads as in Part 1, above, using the 425, 212 and 63- μm sieves and bottom pan. Weigh the fractions to the nearest 0.01 g.

PART 3. RING AND BALL SOFTENING POINT

A. APPARATUS

1. Round 250-mL cans with rims removed
2. An oven capable of maintaining $165 \pm 3^\circ\text{C}$
3. Spatulas or stirring rods
4. Ring and ball apparatus, refer to ASTM Designation: E 28

B. PROCEDURE

1. Perform the test according to ASTM Designation: E 28, Part 8, Procedure for Materials Having Softening Points above 80°C , except as follows: heat approximately 100 g of the material in a 250-mL can for 1 h at 165°C . Remove the can with material from oven, stir rapidly and thoroughly for 10 s with a spatula and carefully fill the rings. Allow the material to cool and proceed.
2. Average the results of two or more tests.

PART 4. REFLECTANCE AND YELLOWNESS INDEX

A. APPARATUS

1. Photovolt or other reflectance meter conforming to ASTM Designation: E 1347
2. Clean tin plates or other clean, smooth surface are required for casting sample disks approximately 50-mm in diameter. Lids for 250-mL cans are satisfactory for this purpose.
3. Round 250-mL cans with inside rim removed
4. An oven, capable of maintaining $165 \pm 3^{\circ}\text{C}$
5. Spatula or other stirring rod

B. PROCEDURE

1. Weigh approximately 100 g of material into the can and heat the contents for 1 to 2 h at 165°C .
2. Remove the can and material from the oven. Stir the contents rapidly and thoroughly for 10 s with the spatula.
3. Cast the material onto the tin plate, or can lid, and allow the sample to cool to room temperature.
4. Follow the manufacturer's direction for use of the Photovolt Reflectance Meter. Using the three tri-stimulus filters, amber (A), blue (B), and green (G) in the Diffuse Reflectance head, #610Y, calibrate each filter with the porcelain enameled secondary standard.
5. Determine the reflectance values of the sample.
6. The daylight luminous reflectance of the sample corresponds to the value obtained with the green (G) tri-stimulus filter. Calculate the Yellowness Index, Y, from the above values for A, B and G using the following equation:

$$Y = (A-B)/G$$

Refer to the Federal Test Method Standard, No. 141 and 6131 for a discussion of the events.

PART 5. TITANIUM DIOXIDE

A. APPARATUS

Unless otherwise stated, all reagents used in the chemical tests shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society:

1. A balance capable of weighing to 0.1 mg
2. A 250-mL beaker or Erlenmeyer flask
3. Hot plate or Meker-type burner
4. Sulfuric acid, concentrated
5. Ammonium hydroxide, concentrated
6. Ammonium sulfate crystals
7. Potassium permanganate solution, KMnO_4 , 0.1 Normal
8. A Jones Reductor, refer to ASTM Designation: D 1394

B. PROCEDURE

1. Accurately weigh 0.5 to 1 g of material from the disk used for the Reflectance and Yellowness Index test into a 250-mL beaker or Erlenmeyer flask.
2. Ignite the sample at 400 to 600°C overnight or long enough to remove the organic matter. Alternately, the sample may be ignited over a burner until all the residue is a pure, light yellow.
3. Determine the TiO_2 content according to ASTM Designation: D 1394, total titanium by the Jones Reductor Method.

4. $\% \text{TiO}_2 = [(\text{mL of KMnO}_4) \times \text{N of KMnO}_4 \times 0.0800] / \text{Sample Weight} \times 100$

PART 6. BOND STRENGTH

A. APPARATUS

1. One pair of concrete blocks, 50 by 90 by 175 mm
2. A compression-type testing machine with a 0 to 2270-kg range fitted with a Bond Test Jig conforming to ASTM Designation: C 321
3. Round 500-mL cans with their rims removed
4. An oven capable of maintaining $105 \pm 2^\circ\text{C}$
5. An oven capable of maintaining $165 \pm 3^\circ\text{C}$
6. Spatulas or other stirring rods

B. PROCEDURE

1. Weigh approximately 300 g of material into the 500-mL cans and heat for 2 h at 165°C .
2. Preheat the blocks for 3 h at 105°C .
3. After heating, remove the blocks from the oven. Immediately remove the can and material from the oven and stir rapidly and thoroughly with a spatula for 10 s.
4. Pour the material onto the 90-mm face of one of the blocks and *immediately* place the other block on top and at right angles so the blocks form a cross, taking care to squeeze the material enough to completely cover the contact surfaces and to form a layer of material approximately 35-mm thick.
5. After the assembly has reached ambient temperature, or allowed to stand overnight, perform the test according to ASTM Designation: C 321. The loading rate shall be 230 kg/min.
6. Calculate the pressure, in Pa, by dividing the total load by the contact area. If the concrete block breaks instead of the bond,

the result is expressed as "in excess of" the particular value obtained.

PART 7. SPECIFY GRAVITY

Determine the specific gravity of the materials according to ASTM Designation: D 153, Method A. Use kerosene as the immersion liquid. Special care must be taken in selecting a representative sample.

PART 8. BLEED-THROUGH ON ASPHALT

Follow the procedures as described in ASTM Designation: D 969 using the equipment for applying the material to make the test panels.

PART 9. COLOR

Samples for color shall be applied with the equipment normally used for application and shall be applied to suitable panels for comparison.

PART 10. SAFETY AND HEALTH

This method may involve hazardous materials, operations, and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Laboratory personnel using this method are exposed to flammable solvents, hot materials, concentrated acids and bases. Prior to handling, testing or disposal of any materials, testers are required to read the Caltrans Laboratory Safety Manual. Users of this method do so at their own risk.

REFERENCES:

ASTM Designations: E 28, E 97, D 1394, D 153, D 969, E 1347 and C 321
Federal Test Method Standard No. 141 and 6131
Caltrans Standard Specifications

End of Test (California Test 437 contains 4 pages)